DURABILITY OF PAVING ASPHALT

PART III

66-18

State of California
Department of Public Works
Division of Highways
Materials and Research Department

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Mr. J. C. Womack
State Highway Engineer
Division of Highways
Sacramento, California

Dear Sir:

Submitted for your consideration is:

FINAL REPORT

on

DURABILITY OF PAVING ASPHALT

Part III

Evaluation of the

Thelen Sphericity Test

for Measuring the Adhesion

Characteristics of Paving Grade Asphalts

in the Presence of Water

Study made by .... Pavement Section
Under general direction of .... E. Zube
Project Supervisor .... J. Skog
Report prepared by .... J. Skog

Very truly yours,

JOHN L. BEATON
Materials and Research Engineer

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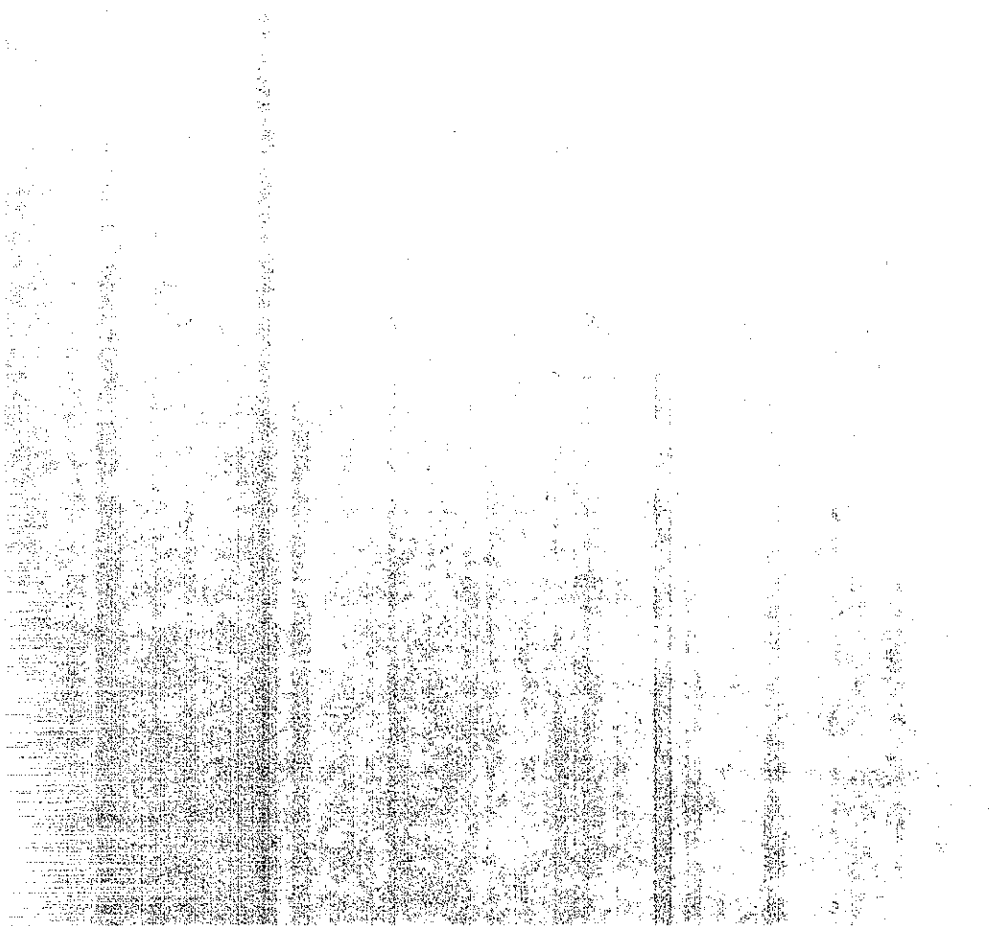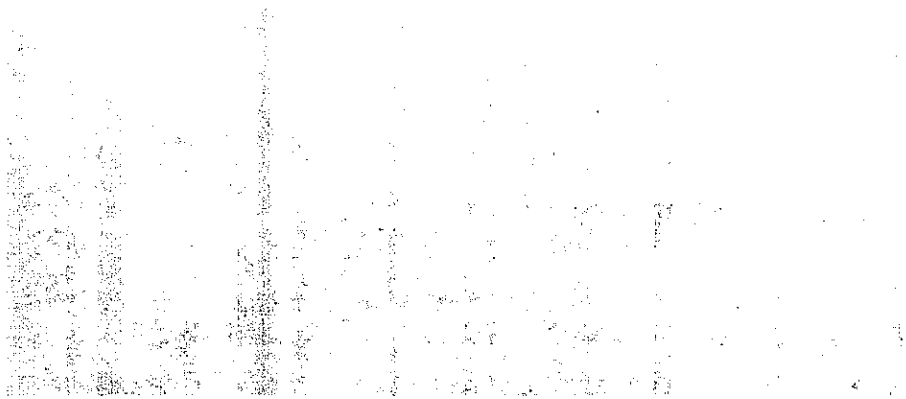
SYNOPSIS

One of the tests developed for routine control of asphaltic binder susceptibility to stripping in the presence of water is the Thelen Sphericity Index. This test measures the degree of adhesion of an asphalt to pyrex glass in the presence of water. A tentative relation was found between the sphericity index, as measured by the test, and the percentage of stripping as measured by the dye stripping technique.

The method did not provide satisfactory repeatability. Further studies of the various factors affecting the test repeatability were performed. However, these studies did not provide repeatable results and the adhesion test was abandoned.

INTRODUCTION

The influence of water on the service performance of bituminous pavements has been the subject of extensive research for many years. Many variables have been studied, and it appears that the most important parameter is the nature of the aggregate surface. However, Gzemski[1] found that the crude source of the asphalt did have a rather pronounced effect on the amount of stripping encountered with a single aggregate source. This study was confirmed by Skog and Zube[2]. Therefore, it seems necessary to develop some form of test that will provide a measure of the binder susceptibility to stripping. This requirement would provide uniformity for any one grade in terms of binder resistance to stripping.

The quantitative dye stripping test[2] together with a reference aggregate could probably be used for control of asphaltic binder susceptibility to stripping in the presence of water. However, the use of a reference aggregate provides complications in attaining a constant source of material, and the test itself is rather complicated for routine control.

E. Thelen[3] devised a test for measuring the stripping coefficient. In this test an aggregate particle is ground to have a smooth surface, and is placed in a glass container. The sample is heated to a certain temperature and a drop of asphalt is placed on the solid surface. It spreads over the ground aggregate surface to form a flat film. The system is now cooled to about 80°C and distilled water is added to the cell containing the aggregate particle. The temperature is maintained for an hour, during which time the asphalt film will strip, if it is not stable in the presence of water. Stripping converts the asphalt film into a ball. The silhouette of this ball is projected on to a sensitized paper, and its contact angle to the stone surface measured. The stripping coefficient is then calculated as shown in the Thelen report.[3]

The Thelen method appeared to offer promise in the development of a routine test for controlling the stripping tendencies of the asphalt. Therefore, a study was initiated to develop a test method and specification requirements. It was proposed to formulate these requirements on the basis of studies involving correlation of the modified Thelen test results with the quantitative dye technique using a number of aggregate sources. Unfortunately, the modified Thelen test method has not furnished consistent results, and this report will only cover the development studies on the test method.

CONCLUSIONS

The method did not provide repeatable results even though studies, described in this report, were performed in an effort to control factors that appeared to have an effect on the test results.

TEST METHOD

The first modification of the Thelen test was the substitution of a pyrex glass plate for the ground aggregate surface. It was believed that a suitably cleaned glass surface would provide a common test surface for measuring asphalt adhesion in the presence of water. Various other modifications were made in order to provide a simple test method for possible use in routine control. The method is outlined as follows: Pyrex glass plates 18 x 30 mm are thoroughly cleaned by a special procedure and dried in an oven at a temperature of 210°F for 30 minutes. Two drops of asphalt are then placed on the plates while in the oven and allowed to spread for a period of 200 seconds. The plates are then placed in a glass cell, 40 x 21 x 80 mm in dimension, and double distilled water at 185°F is added to cover the plates. The cell is then placed in a water bath maintained at 185°F and left for one hour. After this period the cell is removed and placed in a simple photo projector so that the image of the asphalt bubbles may be projected on a piece of white heavy paper. Two methods of measurement are used. The first measures the angle of tangent, and the second measures the sphericity index. As will be shown later, there is an excellent correlation between the angle of tangent and the sphericity index. Therefore, since the sphericity index is simple and more accurate, this measurement was adopted. The index is measured by determining the ratio of the height of the bubble to the width of the bubble. Photographs of the apparatus are shown in Figs. 1, 2, 3 and 4.

TEST RESULTS AND DISCUSSION

Test Procedure Number I is presented in Appendix A. A series of asphalts were tested using this procedure. We noted that samples tended to strip prematurely, thus making it difficult to obtain an asphalt bubble. The results also showed poor repeatability between two separate runs.

Procedure Number II was developed to overcome technical disadvantages found in Procedure Number I. The method II is presented in Appendix B. This method was direct and simple, and operators had no trouble in performing the test.

The tangent angle and sphericity index were determined on a series of asphalts. The results are shown in Table A. There is a very good relation between these two measurements, see Fig. 5, and since the sphericity index is easier to obtain, it was adopted as the unit of measurement.

The values for the sphericity index, shown in Table A, are grouped in Table B to aid in the following discussion:

-4-

There is a marked difference in sphericity index values for separate runs. Each value for a specific run is the average of two individual determinations of the bubble formation on a single plate. These readings were very nearly the same. Therefore, the variation must be caused by variables occurring within the individual runs. Although the individual runs show sizable differences, the results were averaged. The following summarizes our findings based on these results.

1.   There appears to be relation between the sphericity index and the percentage of stripping, see Table C and Fig. 6.

2.   Various asphalt sources show definite differences in sphericity index. Values range from .11 to .96 for the 85-100 grade or from good adhesion to virtually zero adhesion under the same test conditions.

3.   There are definite differences between different samples of 85-100 grade from the same source.

4.   The viscosity of the asphalt does not appear to influence the sphericity index value. Some of the 200-300 grade asphalts have a lower sphericity index, indicating less stripping, than an 85-100 grade from another source.

The results were encouraging and because of the simplicity of the test, it was decided to attempt to control any factors contributing to the lack of repeatability in the test.

The two most important factors that might produce variations between runs are the "cleanness" of the plates and the amount of retained moisture. Thelen,(3), has shown the importance of the adsorbed water layer on the aggregate particle, and the difficulty of removing this film only several molecules thick.

After a number of trials, the following procedure was developed for cleaning and removal of a possible moisture film. The plates were washed in benzene and then dried. Then they were soaked in hot chromic acid (150°F ± 5°F) for a minimum of 14 hours. This was followed by a rinse in hot tap water and boiling in double distilled water in a stainless steel rack. The plates were then placed in an oven at a temperature of 400°F for a minimum of 2 hours. Immediately after this drying period, they were placed in a 210°F oven for 30 minutes and procedure II was then followed for testing.

A series of 85-100 grade asphalts were tested by the new technique and the results showed good repeatability. How-

ever, on rechecking the results at a later date, the results were again found to be erratic.

We believed that an increase in drying time might produce more satisfactory results. Also, it was decided to place two different asphalts on the same plate during testing, in order to study the effect of the plate drying time factor on different asphalts under identical conditions of test. The results are shown below.

| Plate Drying Times | Sphericity Index | | | | | |
| | Plate A | | Plate B | | Plate D | |
	Asph. X	Asph. Y	Asph. X	Asph. Y	Asph. X	Asph. Y
2 hrs. at 400°F plus 1.5 hrs. at 220°F	.77	.95	.72	.95	.72	.95
2 hrs. at 400°F plus 3 hrs. at 220°F	.66	.97	.71	.99	.82	.98
2 hrs. at 400°F plus 4 hrs. at 220°F	.31	.81	.11	.94	.52	.91
2 hrs. at 400°F plus 108 hrs. at 220°F	.17	1.02	.19	.81	.23	.95
24 hrs. at 400°F 1 hr. at 220°F	.16	.90	.15	.78	.19	.95

For any single plate, there is a gain in adhesion with increasing drying times for Asphalt X, but little or no change for Asphalt Y. It appears that a rather long time, high temperature plate drying period would be required to attain an equilibrium condition for Asphalt X. It is apparent that the adhesion relationship of asphalts to carefully cleaned pyrex glass plates is very complex and factors which influence this relation for one asphalt do not apply to another source.

On the basis of these studies, we have decided that the parameters influencing the repeatability of the test are very difficult to control. Therefore, no further work is proposed on this test.

The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Bureau of Public Roads.

REFERENCES

1. Gzemski, F. C., "Factors Affecting Adhesion of Asphalt to Stone", Association of Asphalt Paving Technologists, Vol. 17, p. 74, 1948.

2. Skog, J. and Zube, E., "New Test Methods for Studying the Effect of Water Action on Bituminous Mixtures", Association of Asphalt Paving Technologists, Vol. 32, p. 380, 1963.

3. Thelen, E., "Surface Energy and Adhesion Properties in Asphalt-Aggregate Systems", Highway Research Board Bulletin 192, p. 63, 1958.

TABLE A

Tangent Angle and Sphericity Index for Various Asphalts

Sacto. Res. No.	Source	Grade	Tangent Angle, T, and Sphericity, S							
			Run #1		Run #2		Run #3		Average	
			T°	S	T°	S	T°	S	T°	S
3218	A	85-100	80°	.47	49°	.24	30°	.16	53°	.29
3140	A	85-100	76	.45	23	.11	46	.25	48	.27
3219	A	120-150	76	.39	68	.35	33	.16	59	.30
3217	A	200-300	42	.26	16	.09	66	.37	41	.24
3217	A	200-300	42	.26	16	.09	66	.37	41	.24
3217	A	200-300	21	.08	22	.11	73	.43	39	.21
2787	B	85-100	27°	.12	25°	.11	27°	.14	26°	.12
3016	B	85-100	20	.10	34	.14	24	.11	26	.12
3018	B	200-300	20	.07	15	.07	37	.18	24	.11
3205	C	60-70	155°	.95	145°	.90	161°	.96	154°	.94
2765	C	85-100	125	.79	132	.85	130	.82	129	.82
2790	C	85-100	123	.78	102	.60	131	.80	119	.73
3206	C	85-100	141	.91	156	.98	162	.98	153	.96
3206	C	85-100	141	.91	156	.98	162	.98	153	.96
3206	C	85-100	149	.95	150	.95	164	.98	151	.96
3212	C	120-150	156	.96	154	.93	151	.90	154	.93
2628	C	200-300	83	.46	91	.54	101	.63	92	.54
2792	D	85-100	31°	.15	28°	.12	69°	.36	43°	.21
3210	E	60-70	48°	.24	28°	.15	105°	.62	60°	.34
2793	E	85-100	28	.13	58	.28	102	.59	63	.33
3207	E	85-100	59	.33	29	.13	80	.51	56	.32
3208	E	120-150	29	.11	32	.15	40	.20	34	.15
3190	E	200-300	33	.16	17	.07	55	.28	35	.17
3211	E	200-300	25	.11	25	.11	91	.59	47	.27
3152	F	85-100	33°	.16	71°	.39	35°	.18	46°	.24
2798	G	85-100	66°	.35	83°	.46	74°	.40	74°	.40
3202	H	60-70	33°	.14	23°	.11	47°	.26	34°	.17
2801	H	85-100	22	.09	80	.44	69	.36	57	.30
3200	H	85-100	58	.31	65	.35	20	.09	48	.25
3199	H	120-150	81	.47	15	.08	79	.45	58	.33
3201	H	200-300	18	.09	21	.09	37	.21	25	.13
2807	I	85-100	27°	.13	39°	.18	31°	.13	32°	.15
2651	I	200-300	59	.38	50	.26	71	.38	60	.34
2799	J	85-100	46°	.24	79°	.46	69°	.39	65°	.36

TABLE A (Contd)

Sacto. Res. No.	Source	Grade	Tangent Angle, T, and Sphericity, S							
			Run #1		Run #2		Run #3		Average	
			T°	S	T°	S	T°	S	T°	S
2799	J	85-100	46	.24	79	.46	69	.39	65	.36
2799	J	85-100	97	.56	89	.50	114	.68	100	.58
2804	K	85-100	19°	.08	28°	.12	45°	.16	31°	.13
2812	L	85-100	107°	.57	97°	.57	84°	.50	96°	.55
2812	L	85-100	107	.57	97	.57	84	.50	96	.55
2812	L	85-100	26	.12	36	.19	58	.32	40	.21
2812	L	85-100	22°	.09	76°	.42	101°	.62	66°	.38
3134	M	85-100	20°	.07	84°	.49	61°	.32	55°	.29
3135	N	85-100	77°	.41	85°	.50	15°	.08	59°	.33
3139	O	85-100	34°	.17	98°	.60	114°	.69	82°	.49
3095	P	60-70	76°	.43	70°	.39	91°	.54	79°	.45
3129	O	85-100	76	.43	70	.39	91	.54	79	.45
3129	Q	85-100	119	.72	135	.86	130	.79	128	.79
2440	R	85-100	48°	.26	42°	.26	101°	.65	64°	.39
2441	S	85-100	45°	.22	27°	.12	55°	.29	42°	.21
3175	T	85-100	22°	.09	34°	.16	103°	.65	53°	.30

TABLE B

Sphericity Index for Various Asphalts

Sacto. No.	Source	Grade	Sphericity Index S Run #1	Run #2	Run #3	Average
3218	A	85-100	.47	.24	.16	.29
3140	A	85-100	.45	.11	.25	.27
3219	A	120-150	.39	.35	.16	.30
3217	A	200-300	.26	.09	.37	.24
3217	A	200-300	.26	.09	.37	.24
3217	A	200-300	.08	.11	.43	.21
2787	B	85-100	.12	.11	.14	.12
3016	B	85-100	.10	.14	.11	.12
3018	B	200-300	.07	.07	.18	.11
3205	C	60-70	.95	.90	.96	.94
2765	C	85-100	.79	.85	.82	.82
2790	C	85-100	.78	.60	.80	.73
3206	C	85-100	.91	.98	.98	.96
3206	C	85-100	.91	.98	.98	.96
3206	C	85-100	.95	.95	.98	.96
3212	C	120-150	.96	.93	.90	.93
2628	C	200-300	.46	.54	.63	.54
2792	D	85-100	.15	.12	.36	.21
3210	E	60-70	.24	.15	.62	.34
2793	E	85-100	.13	.28	.59	.33
3207	E	85-100	.33	.13	.51	.32
3208	E	120-150	.11	.15	.20	.15
3190	E	200-300	.16	.07	.28	.17
3211	E	200-300	.11	.11	.59	.27
3152	F	85-100	.16	.39	.18	.24
2798	G	85-100	.35	.46	.40	.40
3202	H	60-70	.14	.11	.26	.17
2801	H	85-100	.09	.44	.36	.30
3200	H	85-100	.31	.35	.09	.25
3199	H	120-150	.47	.08	.45	.33
3201	H	200-300	.09	.09	.21	.13
2807	I	85-100	.13	.18	.13	.15
2651	I	200-300	.38	.26	.38	.34
2799	J	85-100	.24	.46	.39	.36
2799	J	85-100	.56	.50	.68	.58
2804	K	85-100	.08	.12	.16	.13
2812	L	85-100	.57	.57	.50	.55
2812	L	85-100	.57	.57	.50	.55
2812	L	85-100	.12	.19	.32	.21
3134	M	85-100	.09	.42	.62	.38
3135	N	85-100	.07	.49	.32	.29
3139	O	85-100	.41	.50	.08	.33
3095	P	60-70	.17	.60	.69	.49
3129	Q	85-100	.43	.39	.54	.45
3129	Q	85-100	.43	.39	.54	.45
3129	Q	85-100	.72	.86	.79	.79
2440	R	85-100	.26	.26	.65	.39
2441	S	85-100	.22	.12	.29	.21
3175	T	85-100	.09	.16	.65	.30

TABLE C

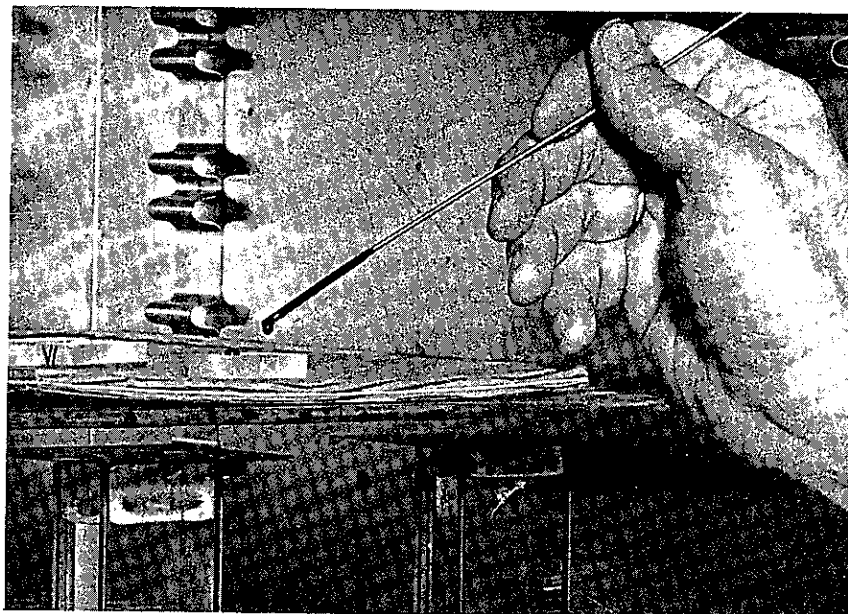### Relation Between Sphericity Index and Percentage of Stripping.

Test Series	Asphalt Code No.	Grade	Stripping* %	Sphericity Index
2	B	85-100	35	.12
2	H	"	37	.27
2	K	"	38	.13
2	E	"	41	.32
2	A	"	44	.28
2	C	"	51	.78

*Determined by Dye Technique, Reference 2, on a single aggregate source.
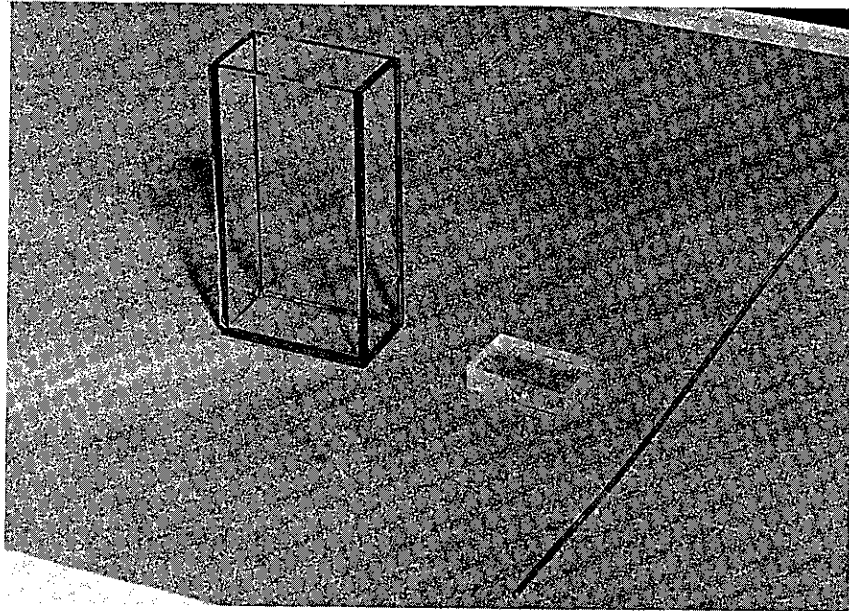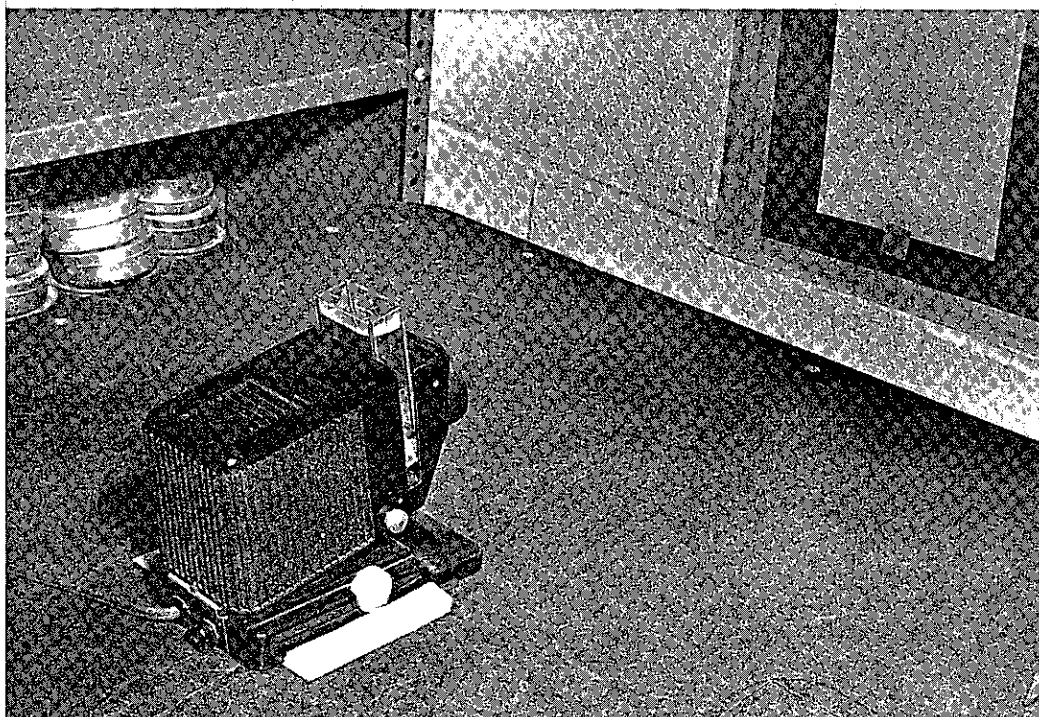
TEST EQUIPMENT

PLACING DROP OF ASPHALT ON PLATE IN OVEN

FIGURE I
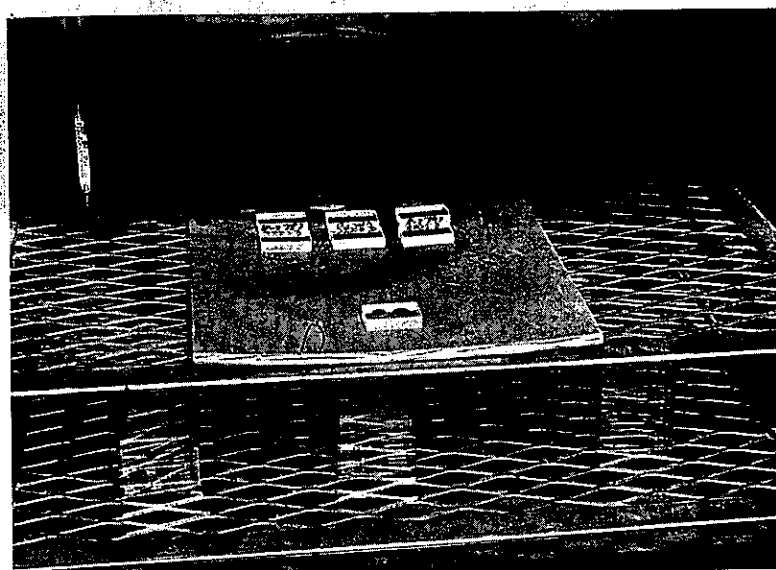
VIEW OF DROPS OF ASPHALT ON PLATE
AFTER 200 SECONDS
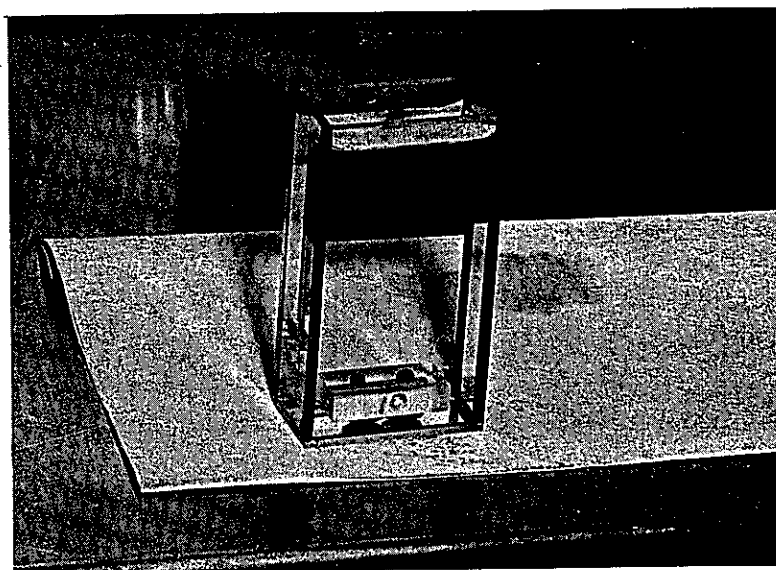
VIEW AFTER PLACING PLATE IN PHOTO CELL

FIGURE 2

VIEW OF PHOTO CELL WITH PLATE IN PROJECTOR

FIGURE 3
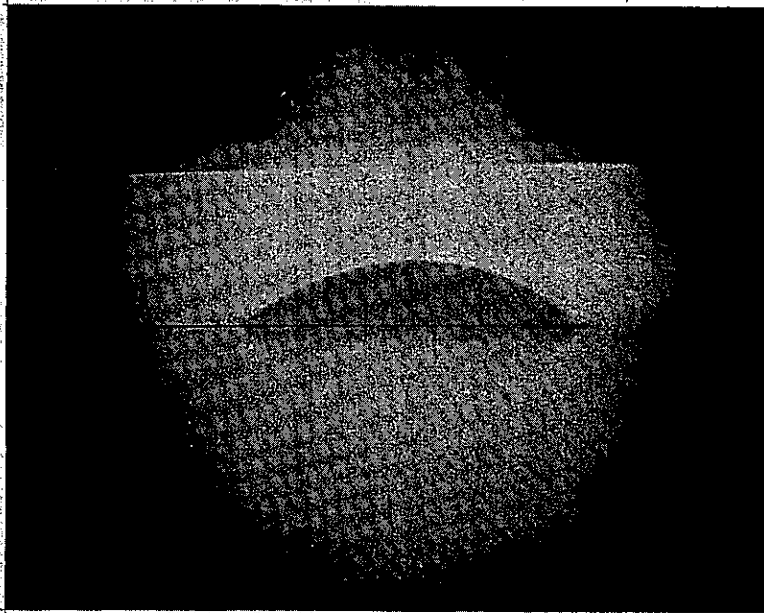
PROJECTED VIEW OF BUBBLE OF ASPHALT WITH GOOD ADHESION
SPHERICITY INDEX = 0.19
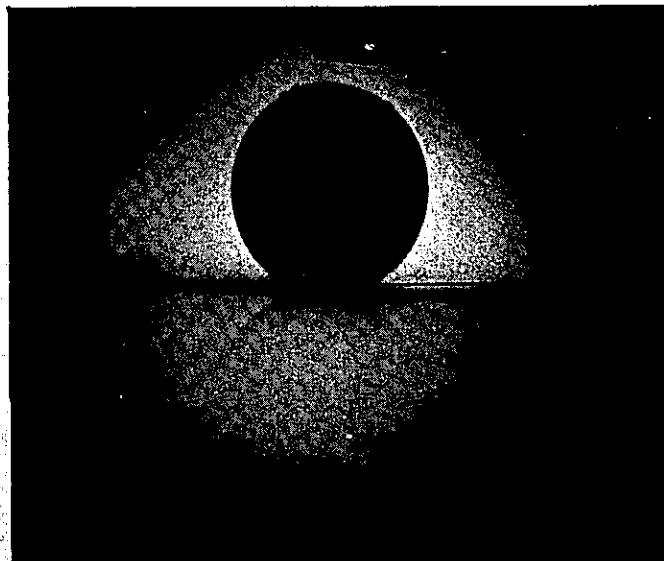
PROJECTED VIEW OF BUBBLE OF ASPHALT OF POOR ADHESION
SPHERICITY INDEX = 1.04

FIGURE 4

CORRELATION OF TANGENT ANGLE AND
SPHERICITY INDEX
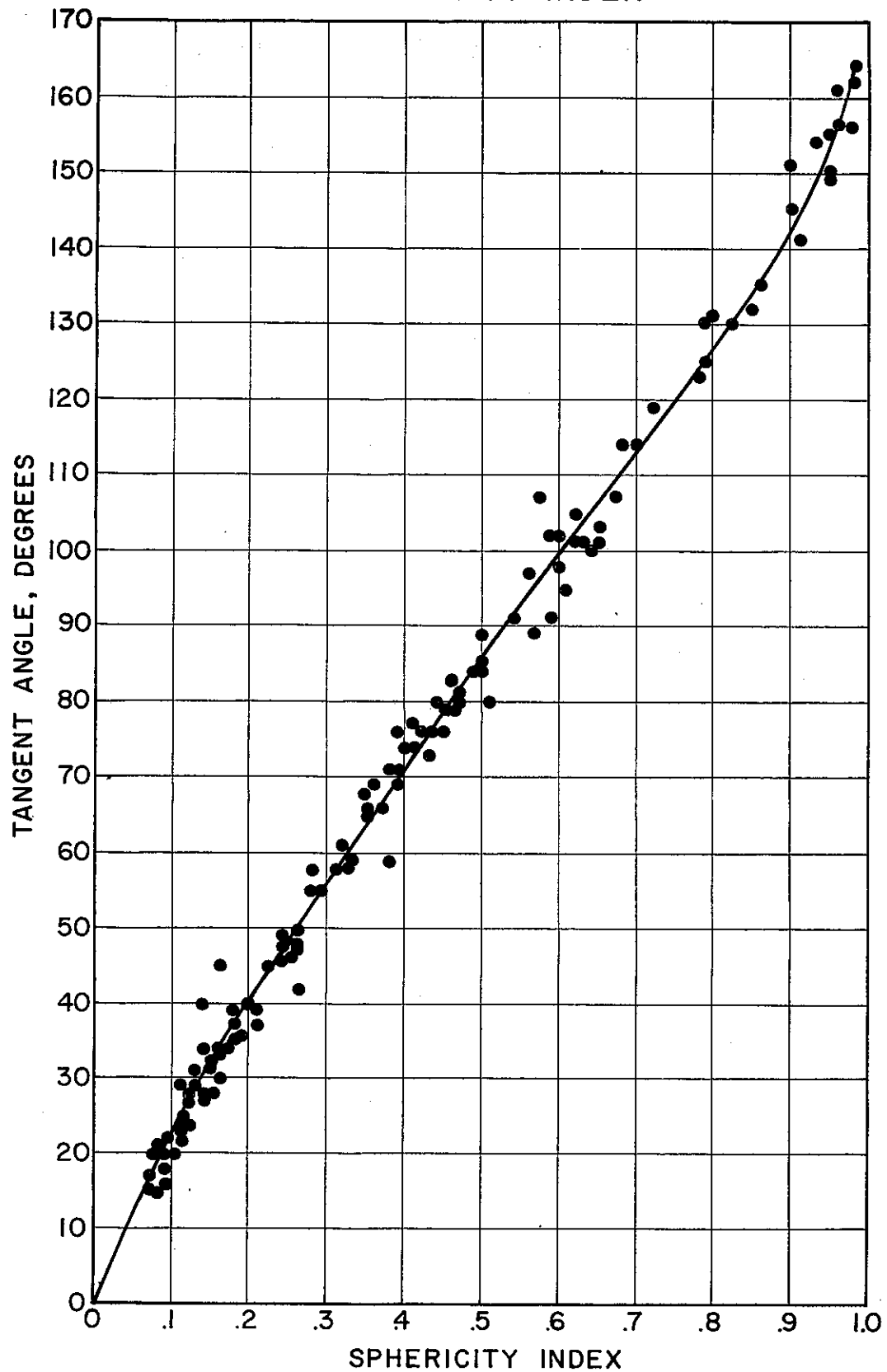
FIGURE 5

RELATION BETWEEN PERCENTAGE OF
STRIPPING AND SPHERICITY INDEX
California 85-100 Grade Asphalts
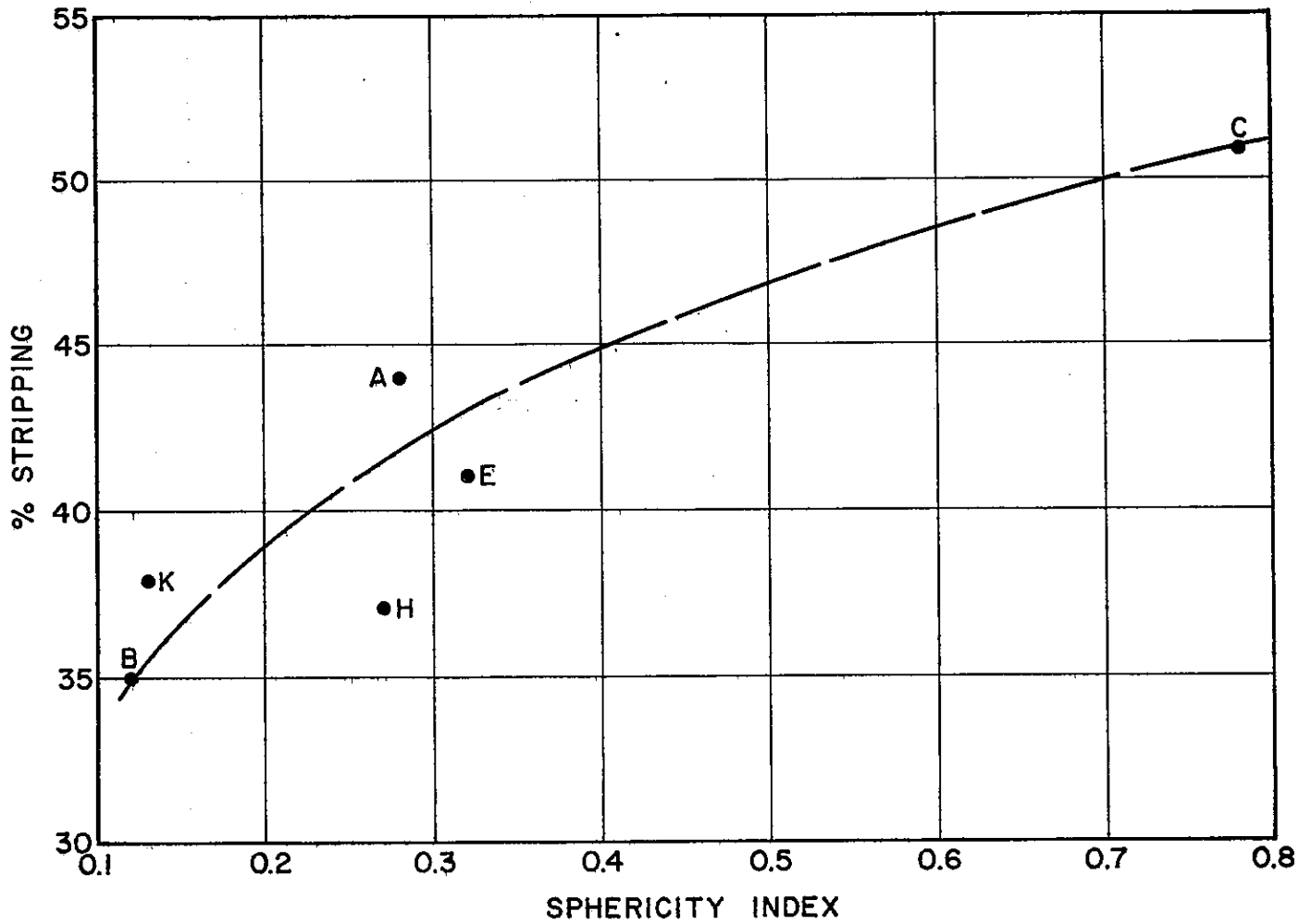
FIGURE 6

Asphalt Adhesion Test

Procedure I

SCOPE:

      To determine the relative adhesion of an asphalt using a pyrex plate as a standard aggregate surface.

I.   Equipment

    1.   Plates - of pyrex plate glass 30 x 18 x 6 mm.

    2.   Glass Cell - cell of plate glass 2 mm. thick, inside dimensions 40 x 21 x 80 mm.

    3.   Rod - of brass, 1-1/2 mm. in diameter x 20 cm.

    4.   Koda Slide Projector - 100 watts.

    5.   Oven - with controls capable of maintaining a temperature of 210°F ± 2°F.

    6.   Oven - with controls capable of maintaining a temperature of 250°F ± 10°F.

    7.   Hot Plate - capable of maintaining a surface temperature of 150°F ± 5°F.

    8.   Pyrex Jar - with lid, about 400 ml. capacity.

II. Procedure

      Wash the plates with benzene and soak in cold chromic acid overnight. Rinse in tap water and place in boiling distilled water for 5 minutes. Dry in an oven for 15 minutes at 250°F.

   Heat the asphalt in an oven until fluid and stir thoroughly.

      Place 2 drops of asphalt on a plate with the brass rod, and allow to remain on a hot plate for 2 minutes.

      Place the plate in a glass cell containing boiled distilled water at 30°C. Then place the cell in a beaker of water also at 30°C.

Heat water in beaker so that water in cell gains in temperature at a rate of 5°C per minute. Continue heating until water in cell reaches 80°C.
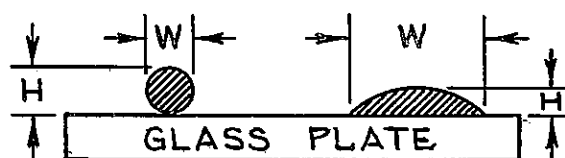
Immediately remove cell and wipe the sides and place it in a slide projector. Measure the tangent angle and sphericity index from the projected image, as shown below.

Tangent Angle
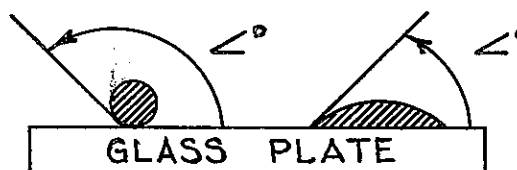
Sphericity Index
$$S = \frac{H}{W}$$

Asphalt Adhesion Test

Procedure II

SCOPE:

      To determine the relative adhesion of an asphalt using a pyrex plate as a standard aggregate surface.

I.   Equipment

    1.   <u>Plates</u> - of pyrex plate glass 30 x 18 x 6 mm.

    2.   <u>Glass Cell</u> - cell of plate glass 2 mm. thick, inside dimensions 40 x 21 x 80 mm.

    3.   <u>Rod</u> - of brass, 1-1/2 mm. in diameter x 20 cm.

    4.   <u>Koda Slide Projector</u> - 100 watts.

    5.   <u>Oven</u> - with controls capable of maintaining a temperature of 210°F ± 2°F.

    6.   <u>Oven</u> - with controls capable of maintaining a temperature of 250°F ± 10°F.

    7.   <u>Hot Plate</u> - capable of maintaining a surface temperature of 150°F ± 5°F.

    8.   <u>Pyrex Jar</u> - with lid, about 400 ml. capacity.

II.   Procedure

      Clean the plates in hot chromic acid (150°F ± 5°F) for not less than 14 hours and then rinse in hot tap water. Boil in a container of distilled water for 15 minutes. Dry in an oven for 15 minutes at 210°F.

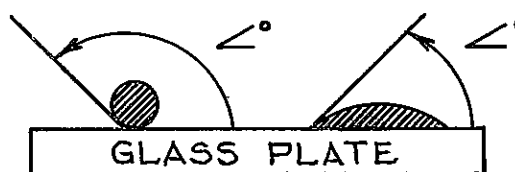   Heat the asphalt in an oven until fluid and stir thoroughly.

      Preheat the plates in a 210°F oven for not less than 30 minutes. Then place 2 drops of asphalt on the plate with the brass rod and allow to spread over the plate for 200 seconds.

      Remove plates from 210°F oven and immediately place in a glass cell containing boiled distilled water at 185°F.

Place the cell in an oven or water bath for one hour at a temperature of 185°F ± 2°F.

Remove the cell and wipe the sides and then immediately place it in a slide projector. Measure the tangent angle and sphericity index from the projected image as shown below.

Tangent Angle

Sphericity Index

$$S = \frac{H}{W}$$